



μ -CTE

Micro-Chamber / Thermal Extractor

Operators Manual

JULY 2006

QUI-1022

VERSION 3.0

MARKES
international

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Micro-Chamber Operators Manual

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1.0 Introduction to the Micro-chamber (μ -CTE)

The Micro-Chamber / Thermal Extractor (μ -CTE™) from Markes International provides industry and researchers with a versatile and automated tool for testing materials.

Key applications include:

- Testing emissions from material surfaces at low temperatures for:
 - correlation with data from conventional emission chambers / cells
 - intercomparison of products within a range (e.g. different colours / patterns)
 - testing prototype, "low-emission" materials
 - monitoring product uniformity in-between formal certification tests
- Testing VOCs and Semi-VOCs (SVOC) in bulk materials for routine quality control (content and emissions testing)
- Testing vapour permeation into and through materials at various temperatures
- Flavour and Fragrance profiling

1.1 Summary of Operation

The system (figure 1) comprises six micro-chambers (up to 28 mm deep and ~45 mm in diameter) which allow surface or bulk emissions to be tested from up to six samples simultaneously. The μ -CTE is compatible with a range of sampling tubes and multiple standard analytical methods.



Figure 1. The Micro-chamber

Conditioned sorbent tubes are attached to each micro-chamber and a controlled flow of air or inert gas is passed through all six chambers simultaneously. VOC and SVOC vapours are swept from the sample material in the micro-chamber and onto the attached sorbent tube. After sample collection, trapped vapours are thermally desorbed and analysed by GC(-MS) as per standard methods.

The thermal desorption analytical process is carried out off-line allowing a fresh set of samples to be introduced to the μ -CTE even while analysis of vapours emitted by the previous set of samples is being performed. This also facilitates chemical analysis by third party laboratories if preferred.

Alternative analysers, combining thermal desorption with process MS or enose detectors, are also applicable in some cases - particularly during quality control of fragrance / odour of foods and consumer products.

2.0 System Installation and Set-up

2.1 Pre-installation

2.1.2 Services Required

2.1.2.1 Power

μ -CTE is automatically compatible with all conventional mains power supplies ranging from 115 to 230 V and 50 or 60 Hz. It is not necessary to manually select or switch voltages. The maximum power consumption of the μ -CTE is 205 W

2.1.2.2 Laboratory requirements

The μ -CTE occupies minimal bench space, (50 cm x 12 cm) and requires sufficient space to allow unhindered access to all of the micro-chambers. Access to the rear of the μ -CTE will be required when changing the gas inlet line (see section 2.3.2).

2.1.2.2.1 Laboratory conditions

As the μ -CTE is a powerful concentrator of VOC's and is often used to determine trace levels of organic analytes, it is advisable to store and operate the μ -CTE in a clean laboratory environment with minimal atmospheric concentrations of organic vapours.

2.1.2.3 Gas supply

The μ -CTE requires a pressure regulated supply of clean air, nitrogen or helium, between the ranges of 10- 60 psi. as a carrier gas through the micro-chambers.

2.1.2.3.1 Cleanliness of gas lines and supply

As the μ -CTE is a concentrator, even trace level contaminants in laboratory gas lines can become significant interferents in the sample obtained. It is recommended that the gas line be constructed of refrigeration-grade copper tubing connected using approved swage-fittings. Laboratory gas line joins and connections must never be brazed. Position the gas supply as close to the μ -CTE gas inlet (Figure 2.) so that the gas lines are as short as possible. Use a high quality, stainless steel diaphragm cylinder head regulator for the gas supply. The cleanliness of the gas and supply can be validated prior to installation of the μ -CTE (see section 2.3.1).

2.2 Unpacking the Micro-Chamber

Remove the instrument from its packaging and inspect the contents. Check every item against the packing list detailed below. Retain the instrument packaging and re-use if ever the system is to be shipped using conventional carriers.

2.2.1 Packing List for Micro-Chamber

The following items are contained in your Micro-Chamber shipment. Please inform your distributor immediately if there are any shortages. Items marked with a * are consumable items and may require replacing at intervals.

Part Number	Description
QUI-1022	Micro-Chamber Users Manual
M-SPC15	Pk 6 Aluminium Collar Spacer 15 mm
M-SPC05 x 2	2 x pk 6 Aluminium Collar Spacer 5 mm
M-DSK15	Pk 24 Aluminium Spacer Disk 1.5 mm
M-MCHOR	Pk 6 Viton O-Rings 032 for Micro-Chamber seal*
M-TCN64 x 2	2 x pk 6 Viton O-Rings 010 for tube receivers*
M-MC006	Pk 6 006 O-rings for detachable lid seal
SERZ-0145	1/8" Copper tubing 3m
SERZ-0024	Mains Cable

2.2.2 Installation - tools required

To complete the installation you will need the following tools:

7/16 wrench / spanner

2.3 Installing the gas lines

Unique technology maintains a constant flow of air or gas through each sample chamber at any given backing pressure, independent of sorbent tube impedance and whether or not a sorbent tube is attached. No pump or mass flow controller is required.

2.3.1 Checking the cleanliness of the gas supply

It is recommended that the gas supply and lines are checked for cleanliness prior to using the μ -CTE. This can be quickly and easily checked by sampling ~ 10 L of gas through a conditioned Tenax tube and then analysing the tube using a

Markes UNITY - GC(-MS/FID) system. Artefact levels of less than 30 ng total compounds are generally acceptable depending on the type of application to be undertaken. If the gas supply is not sufficiently clean then a suitable filter should be placed in-line immediately prior to the μ -CTE. The cleanliness of supply should be re-checked after fitting the filter.

2.3.2 Connecting the gas supply to the μ -CTE inlet

The gas supply should be connected to one of the inlets found on the rear of the μ -CTE (figure 2)



Figure 2. Rear view of μ -CTE

The left-hand inlet is the low-flow gas inlet which allows the user to operate at flows between 10 and 70 mL/min. The right-hand inlet is the high-flow gas inlet which allows the user to operate at flows between 50 and 500 mL/min. In both cases a gas backing pressure of between 10 and 60 psi must be maintained to ensure flow stability of the gas particularly at the lower flow rates.

Once the gas supply is connected, ensure that the unused inlet is capped with the blanking plug supplied.

The flow rate is determined by measuring the flow (with an appropriate flow

meter) from the back of a sorbent tube inserted into the lid of the μ -CTE with the lid closed (figure 3). The choice of sorbent tube used for setting flow rates will ideally be the same type of tube being used during sampling from the μ -CTE. The flow rate is adjusted by regulating the carrier gas pressure. The flow rate can be set with the μ -CTE heated to the desired temperature if required (see section 2.5).

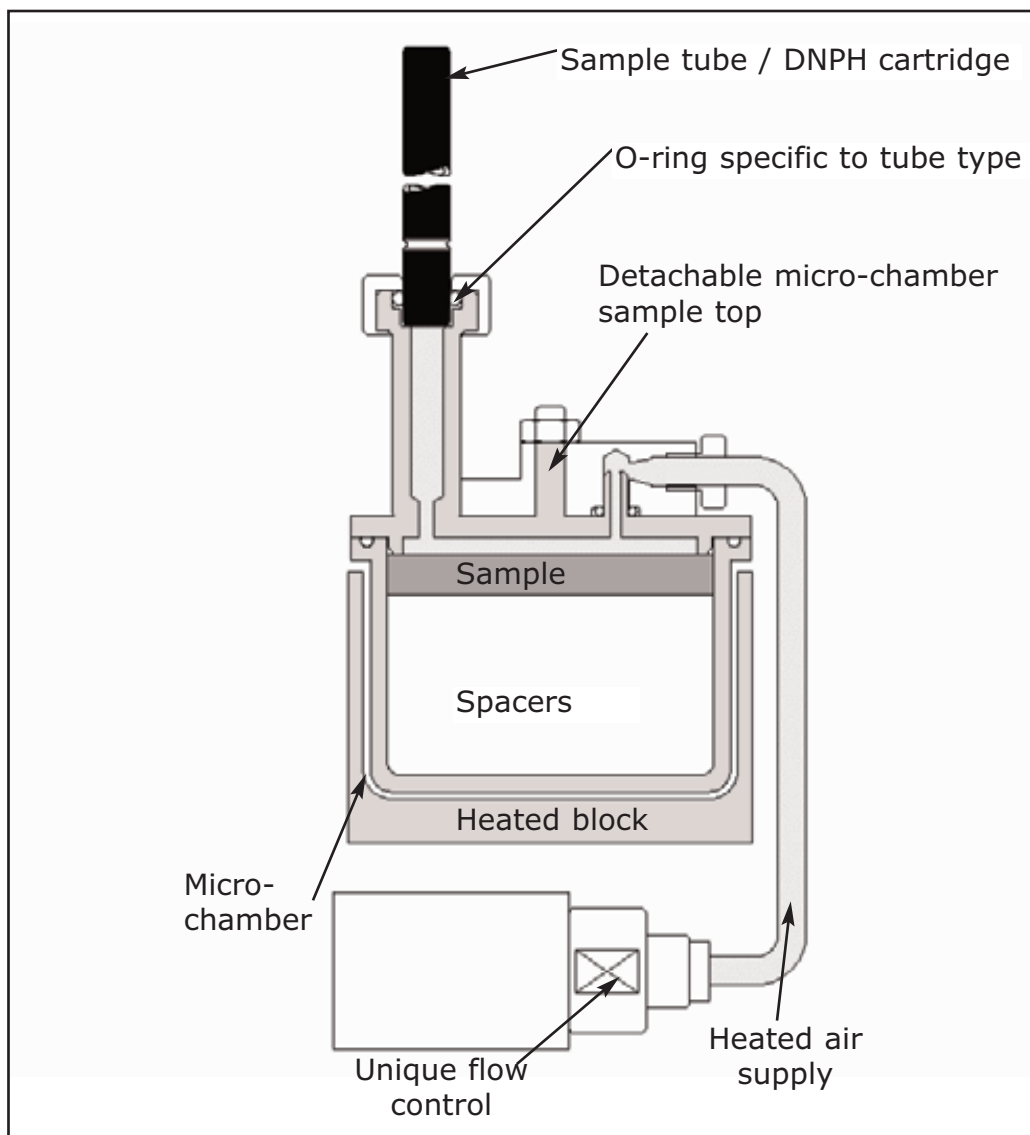


Figure 3. Schematic showing where to put tube

2.4 Connecting the Micro-Chamber

Connect the power cable supplied to the power socket on the rear of the μ -CTE (figure 2) and turn the power switch on.

2.5 Setting the temperature

Independent heating of all μ -CTE chamber components, e.g. sample pans,

chamber lids, air / gas supply tubing, is controlled by the temperature controller on the front panel of the μ -CTE (figure 4).



Figure 4. Front of μ -CTE

Depending on the requirements of the application, micro-chamber sampling can be performed at ambient temperatures or elevated temperatures up to 120°C.

Once the Micro-Chamber is switched on the temperature controller should be lit up and displaying the current temperature set point in green and the current actual temperature of the μ -CTE in red. (figure 5).



Figure 5. Temperature controller

To change the set point push the 'P' button once on the temperature control panel. The red display will change to a flashing 'SP 1' (Set Point 1). While 'SP 1'

is flashing the set point can be adjusted using the up and down arrows on the control panel. To confirm the new set point push '**P**' once more. Alternatively, wait ten seconds after setting the new temperature and the controller will automatically save the new set point. If the temperature is changed, allow sufficient time for the temperature to equilibrate (green and red displays should both show the same figure) before beginning sampling.

3.0 Sampling methods and testing

3.1 Sorbent selection

Regardless of the type of testing (bulk or surface emissions) the gases evolved from the μ -CTE must be collected onto a sorbent tube for subsequent analysis by thermal desorption - GC(-MS/FID). The selection of the sorbent and the conditioning of the tube prior to sampling are critical to the success of the testing as incorrectly selected sorbents may not trap the compounds of interest (or may not release them quantitatively during desorption).

Sorbent selection principally depends upon the volatility (specifically the vapour pressure) of the analyte(s) concerned. In short, the sorbent or series of sorbents selected must quantitatively retain the compounds of interest from the volume of air / gas sampled and must then release those compounds as efficiently as possible when heat is applied and the flow of (desorption) gas reversed. As vapour pressure data is not always readily available, a useful "Rule of Thumb" is to use the boiling point of the component as a guide to its volatility. In general the more volatile the analyte to be trapped - the stronger the sorbent must be. If the analytes to be collected cover a wide boiling point range then it may be necessary to use sorbent tube packed with two or more sorbents in series.

3.1.1 Typical sorbents

There are a large number of sorbents available for use and Appendix 1 gives details of the most commonly used, along with analyte range they are suitable and other information such as maximum temperature etc.

However generally sorbents can be classified as weak, medium or strong as follows:

Weak - porous polymers such as Tenax TA, Tenax GR, graphitised carbon blacks such as Carbograph 2TD, Carbotrap C, Carbo-pack C.

Medium - porous polymers such as the Chromosorb series, Porapak series, HayeSep series, graphitised carbon blacks such as Carbograph 1TD, Carbo-pack B, Carbotrap.

Strong - molecular sieves 13X & 5Å, carbonised molecular sieves such as the Carboxen series, Carbosieve SIII, UniCarb

3.1.2 Conditioning and storage of sorbent tubes

After selecting the correct sorbent tube for the analytes of interest it is essential that the sorbent tubes are correctly conditioned prior to sampling. It is advisable to check the cleanliness of a representative proportion of tubes prior to sampling. Appendix 1 gives further information regarding conditioning temperatures and attainable artefact levels.

Conditioned tubes and sampled sorbent tubes should be capped with $\frac{1}{4}$ inch brass SwageLok type caps fitted with PTFE ferrules. Single sorbent tubes are generally stable for periods of over 1 year at room temperature, however multi-bed tubes should usually be analysed within 1 week of sampling. Storage times longer than 1 week are possible under refrigerated conditions, however in this case care must be taken to ensure that the brass caps remain tight after refrigeration and the tubes must be allowed to equilibrate to room temperature prior to analysis. Further information is given in Markes International Thermal Desorption Technical Support Note 19 - Minimising artefacts - considerations for storage and transportation of sorbent tubes. This can be obtained by registering on the Markes web site www.markes.com

3.2 Bulk emission testing

After setting the required gas flow rate and temperature, materials for bulk emissions testing, (or odour / fragrance profiling), may simply be weighed and placed directly into an empty micro-chamber - or into a custom made inert "sample boat" which is placed directly into the chamber itself.

Allow the sample to equilibrate - typically for 5 - 10 minutes - and then insert a conditioned sample tube into the μ -CTE tube insert to commence collecting emissions. At the end of the sampling interval, remove the tube and cap with brass storage caps as described above.

Sampled tubes are thermally desorbed and analysed by GC (-MS/FID) following standard methods such as ISO 16000-6, ASTM WK 3464, ISO/EN 16017-1, ASTM D6196-03 etc. Alternative analysers, for example systems combining thermal desorption with process MS or enose detectors, are also applicable in some cases.

Note: If sampling at elevated temperatures, sample tubes may be hot when they are removed from the μ -CTE.

Depending on the type of sample material analysed, after sampling the chamber may require cleaning before subsequent use. See Section 4.1 for cleaning techniques.

3.3 Surface emissions testing

Surface emissions testing requires a 4.5 cm diameter circular sample specimen to be cut from the product / material so that it fits snugly into the micro-chamber on top of the supplied spacers. A collar projecting down from the chamber lid helps define both the exposed sample surface area and the depth of the air space above the sample (figure 3). When testing surface emissions 12.82cm² of sample surface area is exposed to the air / gas flow and the air / gas volume above the sample surface is 3.2 cm³.

The μ -CTE is supplied with a complete set of sample spacers which are used to raise the surface of the test sample so that it is flush with the top of the micro-chamber pot. The spacers are placed inside the micro-chamber, underneath the sample, such that the emitting surface is presented to the air flow at the correct height - whatever its original thickness. When using the spring mounted stage (p/n M-SPGSC), ensure that the nuts to the 3 screws are facing down and the stage is supported by at least 1 aluminium spacer ring. Failure to do so will prevent the stage from compressing.

After setting the required gas flow rate and temperature, place the sample inside the micro-chamber and allow the sample to equilibrate - typically for 5 - 10 minutes - and then insert a conditioned sample tube into the μ -CTE tube insert to commence collecting emissions. At the end of the sampling interval, remove the tube and cap with brass storage caps as described above.

Sampled tubes are thermally desorbed and analysed by GC(-MS/FID) following standard methods such as ISO 16000-6, ASTM WK 3464, ISO/EN 16017-1, ASTM D6196-03 etc. Alternative analysers, for example systems combining thermal desorption with process MS or enose detectors, are also applicable in some cases.

Note: If sampling at elevated temperatures, sample tubes may be hot when they are removed from the μ -CTE.

Depending on the type of sample material analysed, after sampling the chamber may require cleaning before subsequent use. See Section 4.1 for cleaning techniques.

4.0 Maintenance

4.1 Cleaning

The μ -CTE (p/n M-CTE100) sample pots, tube inserts and lids are constructed from stainless steel to minimise contamination and carryover effects. However, cleaning of the chamber pots and lids may occasionally be required depending upon the type of sample analysed and the conditions in which they were sampled.

Note: The inert Micro-Chamber (M-CTE100i) has sample pots, tube inserts and lids constructed in inert coated stainless steel to minimise breakdown of labile compounds.

The physical nature of some samples, e.g. viscous compounds, molten polymers etc. will be more prone to contaminating the chamber than more rigid samples. As a result cleaning of the sample pots and lids in between sampling such products may be necessary. For more rigid samples cleaning of the sample pots and lids will be required less frequently and less rigorously.

Having first removed all sealing O-rings, the micro-chamber pots, chamber lids and spacers can be cleaned by one of two methods:

1. The inner surface can be washed with diluted alkaline detergent, followed by two separate rinsings with freshly distilled water. The surface is then

rinsed again with non-denatured ethanol or another appropriate solvent.

2. Inserting all components, with the exception of the chamber lids, into a Vacuum Oven

Individual micro-chamber pots and lids can be removed from the μ -CTE and placed directly into a vacuum oven at an elevated temperature (200-300°C) for approximately 2 hours. Before placing the sample pots into an oven ensure that the O-rings have been removed from the rim of the micro-chamber pots, from the tube connector attached to the lids and from the sample pot lid.

See section 4.2 for further details on O-ring removal.

4.2 Changing O-ring seals

The micro-chamber pot O-ring seal (p/n M-MCHOR (pk 6)) can be found on the top rim of the pot. The tube insert O-ring can be found by unscrewing the retaining cap.

If the O-rings require removal they should be hooked out with the O-ring extraction tool (p/n SERZ-0351) available from Markes International.

New seals should be pushed into position using the O-ring insertion tool (p/n SERZ-0285) available from Markes International, and gently nudged evenly into the seating. Also use the O-ring insertion tool to smooth around the inner diameter of the O-ring as it is being pushed into place to avoid distortion.

Appendix One - Sorbent Selection

What follows is summary information on a selection of the most commonly used sorbents including maximum operating temperatures, recommended conditioning temperatures and typical operating temperatures. Sorbent tubes should typically be conditioned using higher temperatures and faster gas flows than those selected for analysis, however this must **NOT** exceed the temperature limit of the sorbents selected.

When using multi-bed tubes with two, three or even four sorbents it is essential to note the following points:

- sorbents should be packed into the tubes in order of increasing strength, with the weakest sorbent nearest the front (grooved / fritted) end of the tube.
- multi-bed tubes should only contain mixtures of sorbents of similar maximum temperatures otherwise it will not be possible to comprehensively condition the tubes.

Commonly Used Sorbents

Carbograph 2TD (20/40; 40/60; 60/80)

Carbopack C (60/80)

Carbotrap C (20/40)

Sorbent Type:	Graphitised carbon black
Sorbent Strength:	Very weak
Specific Surface Area (m ² /g):	~12
Approx analyte volatility range:	n-C ₈ to n-C ₂₀
Example analytes:	Alkyl benzenes Hydrocarbons to n-C ₂₀
Sorbent Max Temperature:	>400°C
Recommended Conditioning Temperature:	350°C to 400°C
Recommended Desorption Temperature:	300°C to 350°C
Notes:	Hydrophobic Minimal (<0.1 ng) artefacts Some activity with labile compounds Friable

Tenax TA (35/60; 60/80)

Tenax GR (35/60; 60/80)

Sorbent Type:	Porous polymer
Sorbent Strength:	Weak
Specific Surface Area (m ² /g):	~35
Approx analyte volatility range:	n-C ₇ to n-C ₃₀ Bpt. 100°C to 450°C
Example analytes:	Aromatic compounds except benzene

	Apolar compounds bpt > 100°C
	Polar compounds bpt > 150°C
	PAHs and PCBs
Sorbent Max Temperature:	350°C
Recommended Conditioning Temperature:	325°C
Recommended Desorption Temperature:	Up to 300°C
Notes:	Hydrophobic Low inherent (<1 ng) artefacts Inert Graphitised form best for PAHs / PCBs Efficient desorption Use 35/60 mesh to minimise fines and eliminate "leakage" through conventional sorbent retaining gauzes

Carbograph 1TD (20/40; 40/60; 60/80)**Carbopack B (60/80)****Carbotrap (20/40)**

Sorbent Strength:	Medium / Weak
Specific Surface Area (m ² /g):	~100
Approx analyte volatility range:	n-C ₅ to n-C ₁₄
Example Analytes:	Ketones, alcohols, aldehydes & apolar components within the above volatility range Perfluorocarbon tracer gases
Sorbent Maximum Temperature:	>400°C
Recommended Conditioning Temperature:	350°C to 400°C
Recommended Desorption Temperature:	300°C to 350°C
Notes:	Hydrophobic Low artefacts (<0.1 ng) Some activity with labile compounds Friable

Chromosorb 102 (60/80)

Sorbent Strength:	Medium
Specific Surface Area (m ² /g):	~350
Approx analyte volatility range:	Bpt. 50°C to 200°C
Example Analytes:	Alcohols, oxygenated compounds haloforms less volatile than methylene chloride
Sorbent Maximum Temperature:	250°C
Recommended Conditioning Temperature:	225°C (250°C for short (<15 mins) periods only)
Recommended Desorption Temperature:	No higher than 220°C
Notes:	High artefacts (at least 10ng) - for trace level analysis

condition at 225°C and desorb sample tubes no higher than 200°C to reduce background levels

Artefacts generated on storage - for trace level analysis analyse tubes immediately after sampling

Hydrophobic

Inert

Chromosorb 106 (60/80)

Sorbent Strength:

Medium

Specific Surface Area (m²/g):

~750

Approx analyte volatility range:

n-C₅ to n-C₁₂

Bpt. 50°C to 200°C

Example Analytes:

Hydrocarbons, benzene, volatile oxygenated compounds

Sorbent Maximum Temperature:

225°C to 250°C

Recommended Conditioning Temperature:

225°C (250° C for short (<15 mins) periods only)

Recommended Desorption Temperature:

No higher than 200°C

Notes:

High artefacts (at least 10ng) - for trace level analysis condition at 225°C and desorb sample tubes no higher than 200°C to reduce background levels

Artefacts generated on storage - for trace level analysis analyse tubes immediately after sampling

Hydrophobic

Inert

Porapak N (50/80)

Sorbent Strength:

Medium

Specific Surface Area (m²/g):

~300

Approx analyte volatility range:

n-C₅ to n-C₈

Bpt. 50°C to 150°C

Example Analytes:

Volatile nitriles, e.g. acrylonitrile, acetonitrile, propionitrile

Pyridine

Volatile alcohols, ethanol,

Methyl ethyl ketone

Sorbent Maximum Temperature:

190°C

Recommended Conditioning Temperature:

180°C to 190°C

Recommended Desorption Temperature:

No higher than 180°C

Notes:

High artefacts (at least 10ng) - for trace level analysis condition at 180°C and desorb sample tubes no higher than 160°C to reduce background levels

Artefacts generated on storage - for trace level analysis analyse tubes immediately after sampling

Repack after 50 thermal cycles

Hydrophobic
Inert

Porapak Q (50/80)

Sorbent Strength: Medium
 Specific Surface Area (m²/g): ~550
 Approx analyte volatility range: n-C₅ to n-C₁₂
 Boiling point 50°C to 200°C
 Example Analytes: VOCs within volatility range above
 Oxygenated compounds
 Sorbent Maximum Temperature: 250°C
 Recommended Conditioning Temperature: 225°C (250°C for short (<15 mins) periods only)
 Recommended Desorption Temperature: No higher than 225°C
 Notes: High artefacts (at least 10ng) - for trace level analysis condition at 180°C and desorb sample tubes no higher than 160°C to reduce background levels
 Artefacts generated on storage - for trace level analysis analyse tubes immediately after sampling
 Repack after 50 thermal cycles
 Hydrophobic
 Inert

UniCarb (60/80)

Sorbent Strength: Strong
 Specific Surface Area (m²/g): ~1200
 Approx analyte volatility range: C₃ to n-C₈
 Bpt. 30°C to 100°C
 Example Analytes: Very volatile compounds e.g. VCM, ethylene oxide, carbon disulphide, dichloromethane, chloromethane
 Volatile polar compounds e.g. methanol, ethanol, acetone
 Sorbent Maximum Temperature: >400°C
 Recommended Conditioning Temperature: 350°C to 400°C
 N.B. Increase temp from 250°C stepwise and slowly
 Recommended Desorption Temperature: 300°C to 350°C
 Notes: Some hydrophilicity
 Low artefacts (<0.1 ng)
 Excellent batch-to-batch reproducibility
 Inert
 Non-friable

Carbosieve SIII (60/80)

Sorbent Strength: Very Strong
 Specific Surface Area (m²/g): ~800, but primarily operates on molecular sieve principal with 15/40Å pores
 Approx analyte volatility range: Primarily for C₂ hydrocarbons and smaller molecules.

Example Analytes: Bpt. -60°C to 80°C
 Ultra-volatile hydrocarbons
 Sorbent Maximum Temperature: >400°C
 Recommended Conditioning Temperature: 350°C
 N.B. Slow conditioning required as for UniCarb
 Recommended Desorption Temperature: 300°C
 Notes: Some hydrophyllicity
 Low artefacts (<0.1 ng)
 Easily & irreversibly contaminated by higher boiling components - protect with front bed of weaker sorbent

Carboxen 1000 (Range of mesh sizes available)

Sorbent Strength: Very strong for small molecules
 Specific Surface Area (m²/g): >1200,
 Approx analyte volatility range: Permanent gases and light hydrocarbons (C₂, C₃).
 Bpt. -60°C to 80°C
 Example Analytes: Ultra volatile hydrocarbons
 Sorbent Maximum Temperature: >400°C
 Recommended Conditioning Temperature: 350°C
 N.B. Slow conditioning required as for UniCarb
 Recommended Desorption Temperature: To suit analyte
 Notes: Some hydrophyllicity
 Low artefacts (<0.1 ng)
 Easily & irreversibly contaminated by higher boiling components - protect with front bed of weaker sorbent

Molecular Sieve (13X, 5Å)

Sorbent Strength: Very strong for small molecules
 Approx analyte volatility range: Bpt. -60°C to 80°C
 Example Analytes: 1,3 butadiene (13X), nitrous oxide (5Å)
 Sorbent Maximum Temperature: 350°C
 Recommended Conditioning Temperature: 300°C (increase temperature gradually)
 Recommended Desorption Temperature: To suit analyte
 Notes: Significantly hydrophyllic - do not use in humid conditions
 High artefacts (>10 ng)
 Easily & irreversibly contaminated by higher boiling components